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# Analytical-process supercritical fluid extraction: a synergestic combination for solving analytical and laboratory scale problems [1]

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**Identical principles govern the theory and application of supercritical fluid extraction (SFE) whether they are applied in the field of chemical engineering or analytical chemistry. We have used these principles to develop instrumentation and methodology that can be used to solve a wide range of analytical and laboratory problems. The development of larger scale extractors for analytical use will be presented, including modules which allow the extraction of larger samples, multiple samples simultaneously, and highly viscous materials. Key components in the design of these extractors, such as fluid delivery systems, collection devices, and co-solvent addition schemes, will also be described. This equipment and the components have been integrated into a laboratory-wide extraction and processing system.**

## 1. Introduction

Analytical supercritical fluid extraction (SFE) has undergone a rapid evolution since its introduction in the mid-1980's. Early extraction instrumentation was primarily designed by individuals practising supercritical fluid chromatography (SFC), reflecting a trend toward miniaturization consistent with its use as an on-line adjunct for sample introduction in SFC [1]. However, the limitations and problems associated with on-line SFE [2] have accelerated the development and use of off-line SFE to such an extent that it is now the predominant form of SFE that is used by the analytical chemist.

One artifact of the early days of analytical SFE development is the size of the extraction cell or vessel. Cell volumes have tended to remain relatively small (less than 10 ml), partly due to the use of HPLC equipment as extraction vessels, as well as the desire to design instrumentation having a 'small footprint' for the analytical laboratory. Interest in performing larger scale analytical SFE has seen the development of equipment that embraces both the seminal principles and character of process SFE; not only for analytical chemistry, but for the evaluation of supercritical fluid technology in organic chemistry, polymer synthesis, food technology, and chemical engineering [3]. The need to extract a larger quantity of sample not only reflects a concern in obtaining a representative extract from complex and many times heterogeneous samples (that cannot always be easily homogenized [4]), but the desire to obtain a sample large enough for characterization or evaluation after extraction.

At the National Center for Agricultural Utilization Research in Peoria, IL, USA, for the past decade we have been developing several unique devices which address the above problem. Our laboratory routinely performs extractions ranging from milligram quantities of material to over 6 kg on in-line SFE units, bench-scale extractors, and a semi-continuous pilot plant extractor. Such an environment has provided a unique synergism that has permitted us to develop extraction equipment which embraces both the features and scale of process or analytical SFE. Table 1 enumerates several

[1] Names are necessary to report factually on available data; however the USDA neither guarantees nor warrants the standard of the product, and the use of the name by USDA implies no approval of the product to the exclusion of others that may also be suitable.

Table 1
Applications of laboratory-scale supercritical fluid techniques (NCAUR)

1. Low cost, high sample capacity SFE
   Widespread use with respect to sample types including: meats, seeds, food products, mycotoxin-contaminated grains, adsorbents, oil–water emulsions, fungal cultures
   Typical conditions: sample size = 10–500 g, cell volume = 10–140 ml (one cell), pressure = 17–105 MPa, temperature = 40–200°C, $CO_2$ flow-rate = 1–20 l/min (expanded)
2. Simultaneous multiple sample extraction
   Pesticide and fat extractions from a variety of food and agricultural products including seeds, meats, snack foods, milk
   Typical conditions: sample size = 15–20 g/cell, cell volume = 70–100 ml/cell, pressure = 35–70 MPa, temperature = 40–80°C, $CO_2$ flow-rate = 4–5 l/min (expanded)
3. SFE/supercritical fluid reaction
   Utilized for transesterification reactions involving enzymatic or gas–solid catalytic conversion of vegetable oils, fats from meats, oilseeds
   Typical conditions: sample size = 15–20 g, cell volume = 52 ml, pressure = 17 MPa, temperature = 50°C, $CO_2$ flow-rate = 8 l/min (expanded), reactant addition flow-rate = 0.01 ml/min
   Reactor conditions: same as above, except cell size 5.1 ml packed with 1.4 g lipase catalyst
4. SFE/co-solvent addition
   Phospholipid extraction/fractionation from oil seeds, nutritional labeling fat analysis from food samples, aflatoxins from grains, taxol from yew wood
   Typical conditions: sample size = 50 g, cell volume = 115 ml, pressure = 35–70 MPa, temperature = 80–150°C, $CO_2$ flow-rate = 5 l/min (expanded), cosolvent flow-rate = 0.4–2.7 ml/min
5. Preparative SFE/SFC
   Tocopherol concentrates from rice bran, soya flakes wheat germ, barley bran
   Typical conditions (SFE): sample size = 70 g, cell volume = 140 ml, pressure = 25–70 MPa, temperature = 40–80°C, $CO_2$ flow-rate = 5 l/min (expanded)
   Typical conditions (SFC): chromatographic packing = 16 g silica gel (60/200 mesh), cell volume = 70 ml, pressure = 25–70 MPa, temperature = 40–80°C, $CO_2$ flow-rate = 5 l/min (expanded)

of these larger-scale units which have been fabricated for specific purposes.

In this contribution, we shall discuss primarily the design and developmental philosophy behind such instrumentation. Results that have been obtained on such equipment will only be mentioned sparingly, since they have been documented in the literature. Emphasis instead will be placed on illustrating the versatility of such units noted in Table 1. Finally, multi-sample, multi-pump units will be advocated for servicing the needs of analytical chemists and a design is given for implementing such instrumentation on a laboratory-wide basis, employing a centralized $CO_2$ distribution system.

## 2. The basic extractor system

Fig. 1 illustrates the basic extractor design which we have successfully utilized in our laboratories for over 15 years. This unit was initially designed to study the SFE of seed oils [5], but was incorporated into our analytical SFE program in the late 1980s to extract fat and pesticides from food products [6]. The unit has been traditionally serviced by a gas booster unit (C), delivering pressurized gaseous $CO_2$ from a cylinder (A). This compressor can be obtained from Haskel (Burbank, CA, USA), and is their Model AGT 62/152. This particular booster is quite satisfactory for SFE, allowing the analyst to reach pressure in excess of 70 MPa while delivering the high flow-rates required for processing larger samples.

Carbon dioxide is delivered without heat tracing to an oven enclosure (dotted line) and can be diverted downwards or upwards to a vertically-held

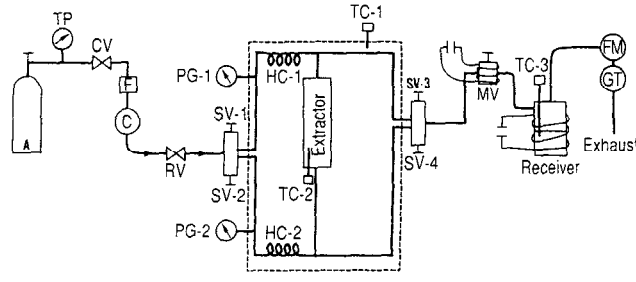


Fig. 1. Generic laboratory SFE unit. A = $CO_2$ cylinder; TP = cylinder pressure gauge; CV = check valve; F = filter; C = air-driven gas booster compressor; RV = relief valve; SV = on/off switching valve; PG = pressure gauge; HC = equilibration coil; TC = thermocouple; MV = micrometering valve; FM = flow meter; GT = gas totalizer.

extractor by using a double switching valve (SV-1, SV-2). Conversion to the supercritical state is achieved via a generous length of helical coil (HC-1, HC-2) before starting the extraction. Extraction cells have been fabricated within our laboratory from 316 stainless-steel tubing usually having lengths of 30.5 or 61 cm. Depending on the wall thickness, such extraction vessels can hold 50–70 or 100–140 ml of material for 70 or 140 MPa extractions, respectively. We have employed up to eight of these vessels in series to affect the extraction of a large quantity of material, using a discarded gas chromatographic oven to hold the multiple extraction vessels. Individual extraction vessels have an almost infinite lifetime, due to the use of a unique self-sealing closure [7] that uses no polymeric component in sealing the vessel under pressure.

Extracts are conveyed out of the extractor through another dual switching valve (SV-3, SV-4) into a heated micrometering valve. This valve must be heated to counteract the effects of Joule-Thomson cooling caused by depressurization of $CO_2$. We have accomplished this by various methods, including heating tapes, hot air dryers, and cartridge heaters in aluminum blocks encasing the micrometering valve. Collection of the supercritical fluid extracts has been accomplished via a number of devices, including bolted closure autoclaves, glass round bottom flasks, and sorbent-filled collection tubes. In Fig. 1, a specially-designed bolted autoclave was used to collect viscous seed oil samples or fats. This component had provision for periodical sample withdrawal via a tube inserted into the bottom of the receiver vessel. Expanded gas was conveyed from the receiver through a crude rotameter (FM) and into a gas totalizer (dry test meter).

Use of this basic and relatively inexpensive SFE unit is well documented in the literature [8–10]. The booster unit reacts to a downstream loss in the system pressure, however typical pressure losses are only 1% of the gauge reading. Likewise, flow is also relatively constant, however, we routinely run extractions to a fixed volume or mass of fluid to assure reproducibility. A commercial analog of this unit is now available and is offered by Applied Separations (Allentown, PA, USA).

## 3. Variations of the basic extractor unit

The unit described in Section 2 is very adaptable to modification and several variations of the basic

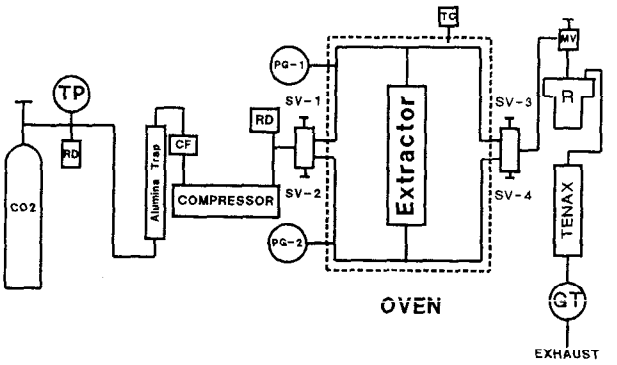


Fig. 2. SFE unit for collection of volatiles and non-volatiles. RD = pressure relief disk; R = receiver. All other symbols same as Fig. 1.

design shown in Fig. 1 have been used in performing SFE in our laboratory. Fig. 2 depicts one of these variations, designed primarily to capture volatile components from seed oils [11], using off-line SFE. Comparison with Fig. 1 shows that the basic flow schematic and many of the unit's components are identical, but several additional features have been added to enhance the performance of the module.

Historically, our extraction units have utilized welding-grade $CO_2$ to minimize the expense associated with performing larger scale analytical SFE. As reported previously [12], this fluid source can be 'cleaned up' substantially with the use of Alumina C, placed in a tubular trap, after the gas cylinder and before the compressor. A superior cleanup sorbent has also been used by Hopper et al. [13], consisting of a 1:2 mixture of coconut charcoal–Alumina C.

In addition, the sample collection scheme has been modified to include not only the previously mentioned bolted autoclave (R), (to capture non-volatile coextractives such as triglycerides), but volatile species as well, on a preconditioned sorbent trap consisting of Tenax. Studies must be conducted to assure that the desired volatiles are efficiently trapped on the sorbent at ambient conditions [14] to avoid breakthrough of the volatiles from the Tenax. An alternative to the above methodology has been developed in our laboratory and features on-line SFE with selective removal of volatile components from seed and meat matrices followed by GC–MS analysis [15]. Unfortunately the samples used in this procedure are only 0.5 g.

A variation of the basic extractor unit has also been designed to elucidate the effect of a co-solvent on the SFE. Initially, a unit which incorporated a

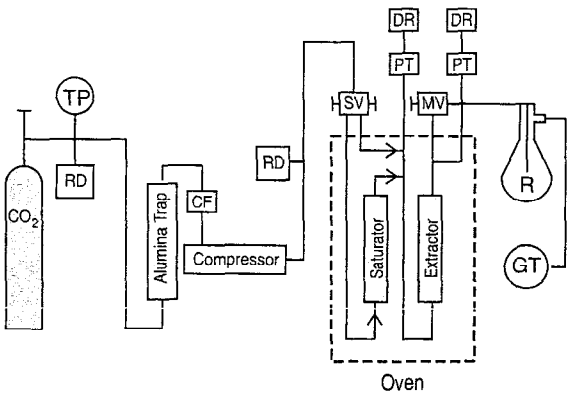


Fig. 3. Ultra-high pressure SFE with co-solvent saturator. PT = pressure transducer; DR = digital readout. All other symbols same as in Figs. 1 and 2.

co-solvent presaturator vessel having dimensions similar to the previously described extraction cells was utilized. The chosen co-solvent is suspended on glass wool or a similar inert sorbent and introduced to the extraction vessel by passing the compressed fluid through the saturator vessel prior to entrance into the extractor vessel. As noted in Fig. 3, a dual switching valve (SV) is used to allow initial extraction of the sample with neat supercritical (SC)-$CO_2$, before initiating the SC-$CO_2$/ co-solvent extraction step. The use of round bottom flasks as receiver (R) vessels has proven advantageous with this unit. Such flasks are relatively cheap and allow multiple collections of discrete fractions of the extract as well as examination of the extract after extraction in the presence of the co-solvent. Use of a fixed co-solvent saturator vessel does not impose any pressure limitations on the use of the extractor, provided that all of the necessary components have appropriate pressure ratings. The unit shown in Fig. 3 was designed to perform extractions up to 105 MPa.

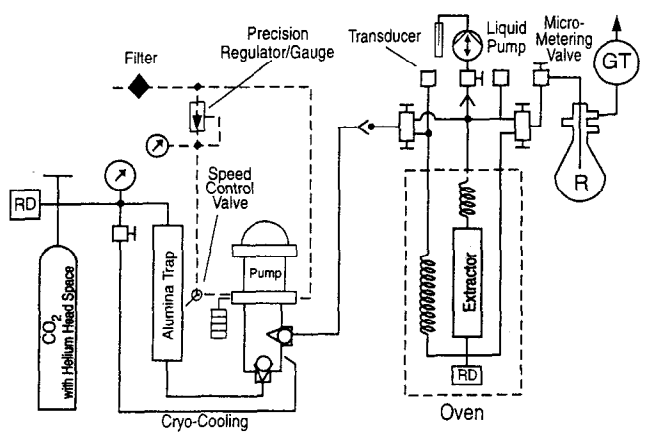


Fig. 4. Co-solvent addition module for SFE and SFR. All symbols as previously defined.

A more sophisticated and slightly more expensive unit for adding a co-solvent to the supercritical fluid is presented in Fig. 4. Here the booster compressor has been replaced by a liquid pump, requiring sub-ambient cooling for efficient operation. Cooling is accomplished by wrapping coils around the pump head and circulating coolant from a thermoregulated circulating bath. To assist in the liquefaction of the extraction fluid, we have used helium headspace liquid $CO_2$ cylinders, pressurized to at least 13.8 MPa. This extra precaution avoids any cavitation of the fluid at the pump head and ensures an even, and energy efficient, delivery of the fluid to the extractor vessel.

As depicted in Fig. 4, co-solvent is delivered continuously with the assistance of a liquid pump. Since many conventional liquid chromatography pumps are rated for operation only at 41.4 MPa, we have employed an older Beckman 100A pump to allow us to deliver co-solvent against a 70 MPa backpressure. The valving arrangement in Fig. 4 allows initial extraction with neat SC-$CO_2$, followed by co-solvent addition to the $CO_2$. Addition of the co-solvent/SC-$CO_2$ mixture to the extractor vessel is from the top down to avoid any buoyancy effects associated with the mixture. Note that the SC-$CO_2$/co-solvent is equilibrated in a coil after addition of the solvent to the $CO_2$. A check valve (Part No. SWO 2200, Autocalve Engineers, Erie, PA, USA) is provided after the co-solvent pump to prevent the passage of fluid back into the liquid pump. The unit has proven very versatile in numerous studies at NCAUR, including the extraction of taxol and to affect enzymatic conversions of triglycerides to their methyl esters (FAMES) for analytical purposes.

## 4. Multi-pump and -sample systems

To increase the versatility of SFE in our laboratory, we have developed multi-pump and -sample systems. We routinely incorporate a multi-pump array to service a single extractor in order to:

- provide more accurate control over specific pressure and flow rate ranges,
- test the delivery of extraction fluids from various pumps/compressors and storage cylinders, and
- provide quick interchange to properly match the pumping unit to the task at hand.

Fig. 5 shows one of these pump arrays servicing

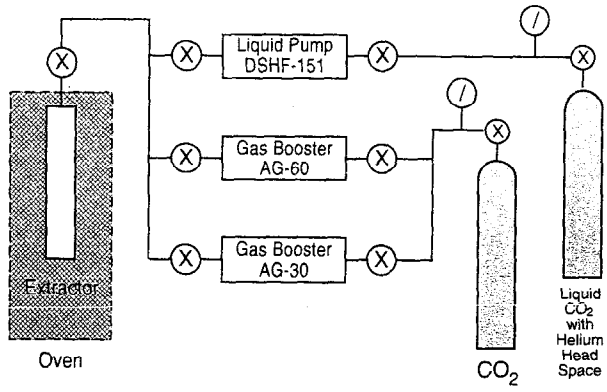


Fig. 5. Multiplex fluid delivery system

one extractor module. Selection of pumps or compressors is made according to the above criteria. Fig. 5 incorporates two Haskel gas boosters and one Haskel liquid pump. The Model DSHF-151 unit was selected to pump liquified $CO_2$ at pressures up to 172 MPa, while Models AG-30 and AG-62 are single-stage (ended) compressors capable of delivering fluids at 31 MPa and 62 MPa, respectively. The AG-30 model is to be preferred in the lower pressure range rather than the AG-62 since it permits more accurate control of the delivery pressure of the extraction fluid. This system has been recently utilized to measure the effect of a helium headspace $CO_2$ source on the solubility of oils in SC-$CO_2$ [16].

Fig. 6 shows one method of delivering liquified carbon dioxide capped by helium headspace to the above mentioned liquid pump. The most efficient fill at the pump head can be realized by precooling the carbon dioxide prior to its entering the pump. This precaution coupled with pump head cooling via Joule-Thomson expansion of a $CO_2$ jet provides an excellent fill and avoidance of cavitation at the pump head. Carbon dioxide is sprayed on the pump

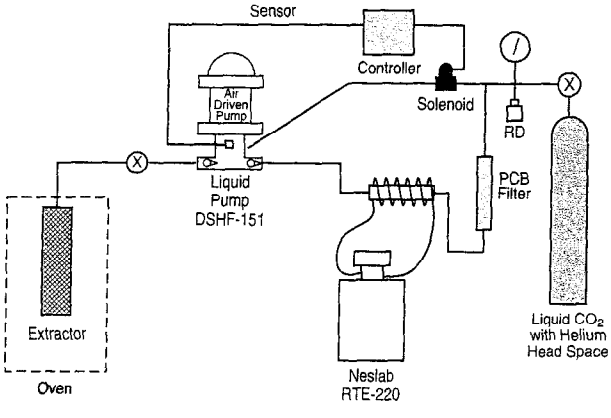


Fig. 6. Liquid booster pump cooling system

head by demand using a controller circuit consisting of a thermocouple attached to the pump head in series with a controller which activates a solenoid valve. The source of coolant can come from a separate $CO_2$ cylinder or be obtained from the $CO_2$ cylinder used to provide fluid to the extractor proper.

Our laboratory has pioneered the development of simultaneous multi-sample SFE. The basic principle behind this technique is the simultaneous extraction of *n* samples in a parallel mode. This operation required the construction of an apparatus that could provide simultaneous extraction of six samples in parallel. Several prototypes have been developed for this purpose [13,17]. Fig. 7 is the flow schematic of the apparatus. Careful inspection of this diagram will reveal that the basis of the unit is a repetition of the basic unit described in Section 2.

The fluid delivery system is very similar to that just described. A series of flow control and shutoff valves, operating in series, provides *manually adjustable* and *stable* flow-rates to each of six extraction vessels. Flow is primarily controlled by the addition of a micrometering valves after each extraction vessel (see Fig. 7). A two-stem valve is inserted before the micrometering valve as a diagnostic device — to relieve pressure from the column if required or to measure fluid flow. A novel flow restrictor, developed by Hopper [13], is also inserted before the extract is totally decompressed to avoid formation of a volatile aerosol and erratic deposition of the extracted analytes into the collection vessels (flasks). Note that both the collection and extractor vessels have their own ovens, juxtaposed to house each array of vessels in its own thermally-controlled environment.

The described instrumentation was specifically designed to process large samples mandated in established regulatory protocols [18,19]. Many of these samples are in the 25–50 g range, thereby requiring the size vessels described above. In addition, the use of extraction enhancers, such as Hydromatrix [20], or in-situ sample cleanup sorbents [21] in the extraction vessel proper, justify the need for larger extraction vessels. By subtly redesigning the unit described in Fig. 7, it would be possible to run simultaneously extractions at different pressures, thereby saving the time associated with multiple runs performed on conventional SFE equipment. It should be noted that a

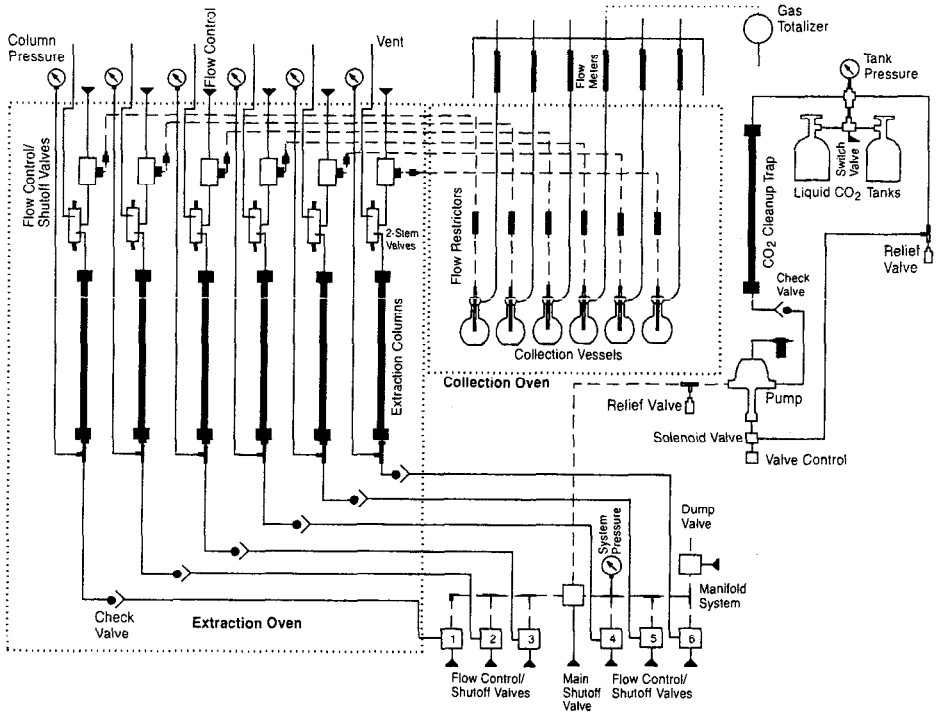


Fig. 7. Simultaneous multiple sample SFE unit.

minaturized analogue, utilizing the above approach for multi-sample extraction, is commercially available. (Model 703, Dionex, Sunnyvale, CA, USA).

## 5. Jet extractor for viscous samples

Many of the systems and components previously described can be utilized to construct special extraction systems for samples which are difficult to process using conventional modes of SFE. Fig. 8 illustrates the design of a 'jet' extractor for processing highly viscous samples containing an extractable component. This unit is based on the system described by Stahl [22] for deoiling lecithin concentrates. The unit can be conveniently assembled on a metal flexaframe support so as to accommodate the height of the apparatus. The solids reservoir and two collector vessels were each $30.5 \times 2.54$ cm, 316 stainless-steel high pressure tubing. The sample to be extracted is placed in the solids reservoir and extruded into the jet tube assembly by a compressed $N_2$ 'pushing' gas whose flow-rate is controlled by a micrometering valve (MV).

Within the pictured three-way valve, it meets a stream of SC-$CO_2$, delivered by a system similar to that used for the pusher gas. It is critical in the three-way valve that the viscous sample be injected through the specified 0.16 cm capillary into a larger concentric tube to avoid viscous backstreaming and ensure adequate contact with the SC-$CO_2$. The solubilized component is then routed through a back-pressure relief valve (BPRV, Haskel Part No. 15700-26), where it undergoes decompression, thereby precipitating the extracted component in the liquid collector.

The extracted sample then falls into the solids collector and can be extruded through the on/off valve at the bottom of this vessel for collection.

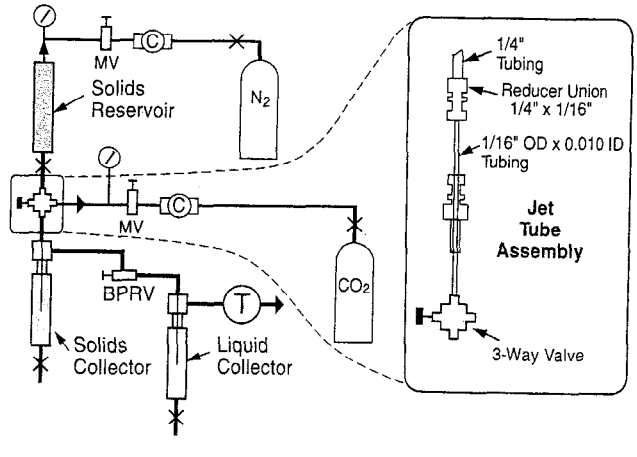


Fig. 8. Laboratory scale jet extractor system. BPRV = back pressure relief valve; T = gas totalizer. All other symbols as previously defined.

We have used the above unit to deoil lecithin, resulting in the extraction of a triglyceride-based oil and the collection of a deoiled phospholipid mixtures in the solids collector. Careful control must be exercised over the relative flow-rates of the pushing and extraction gases so as to maximize the contact time between the sample and the extraction fluid. Unfortunately, in a laboratory scale apparatus one cannot use long contacting tubes that have been utilized in engineering scale deoiling of lecithin concentrates [23]. However, the described system has been successfully used for the preparation of samples that require defatting before instrumental analysis.

## 6. A laboratory fluid delivery system

During the development of the above-described systems, it became imperative for us to develop a laboratory-wide gas distribution system which would allow different extractors to be connected to various compressor or pumps. Fig. 9 illustrates the system which permitted the distribution of $CO_2$ to five extraction units dispersed over three rooms. High pressure tubing was used to connect the four pumps located in different rooms to the designated extractors. A series of on/off valves (S-1 through S-13) were used to isolate or combine certain pump/extractor combinations, depending on the instrumentation needs of a particular experiment. For example, if we desired to use booster compressor AG-30 (C-2) with Extractor 2 in a different room, valves S-8, S-6, S-4, and S-3 would be closed and valves S-7, S-5, and S-4 would be opened. We have used the depicted system for over four years at NCAUR and it has saved us considerable time and expense in reconfiguring extraction instrumentation.

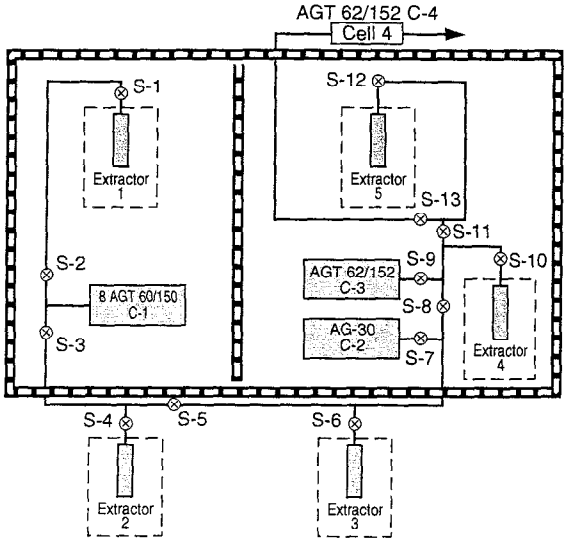


Fig. 9. Integrated laboratory system for supercritical fluid techniques. S = on/off valve; C = booster compressor.

The above scheme suggests that a laboratory of the future doing a high volume of analytical SFEs would have $CO_2$ piped in as a normal utility, similar to the availability of compressed air, natural gas, or vacuum in current laboratories. Hence a central storage tank, similar to those placed outside a building, would be the source of $CO_2$ for SFE. Such an arrangement would save on the storage, cost, and demurrage of multiple cylinder arrays which currently service many laboratories.

## 7. Conclusions

The concepts discussed here suggest that supercritical fluids can be integrated for widespread use in a laboratory environment by combining developments from both process- and analytical-scale experimentation. Such a development opens up the use of supercritical fluids for both research and routine use by not only analytical chemists, but scientists in other technical fields.

For the analytical chemist specifically, larger scale SFE offers several benefits. First, it can enhance the precision of the extraction, which has been shown to be a function of the sample size for samples even under 10 g [24]. Secondly, it can provide a ready enhancement in the scale of the extraction should the results from the analytical SFE prove of interest to engineers, synthetic chemists, etc. And thirdly, it readily accommodates sample sizes associated with traditional analytical protocols (Soxhlet extraction), easing the acceptance of the technique by analytical chemists and its integration into the analytical laboratory.

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